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16. ABSTRACT

In 1975 the U.S. Environmental Protection Agency distributed calibrated solutions of polonium-210 to laboratories interested in participating in an intercomparison study of polonium-210 analysis. Participants were asked to perform a quantitative radioactivity analysis of the solution. The results reported by all the participating laboratories are given here. Fifty-four percent of the reported activity values were within ± 10 percent of the activity value certified by the National Bureau of Standards.

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INTERLABORATORY INTERCOMPARISON OF POLONIUM-210 MEASUREMENTS

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INTRODUCTION

The Quality Assurance Branch at the Environmental Monitoring and Support Laboratory in Las Vegas, Nevada, has maintained an active interlaboratory intercomparison study of radioactivity measurements of environmental media for the last 3 years (1,2,3). Over one hundred State laboratories, Federal laboratories, commercial environmental monitoring laboratories, national laboratories of other countries, nuclear power facilities, and university laboratories have participated in these studies during this time.

The Quality Assurance Branch has also distributed, upon request, calibrated samples of selected radionuclides to these laboratories for use in calibrating their instruments and evaluating their analytical techniques.

In October 1975 the Radioactivity Section of the National Bureau of Standards delivered 40 alpha-particle test solutions of polonium-210 to the Quality Assurance Branch. They also supplied a Report of Calibration for these solutions (Appendix A).

Thirty-nine test solutions were sent to laboratories which have used EPA's quality assurance services in the past. These laboratories agreed to analyze the test solutions for activity and purity and return a test report form similar to that used by the National Bureau of Standards (NBS) (4). Twenty-four of these laboratories did return the test report; five more indicated that they were unable to perform the analysis during the time requested. In April, Reports of Calibration were sent to those which had received the test solutions. The participants are listed in Table 1.

TABLE 1. LIST OF PARTICIPANTS, ALPHA-PARTICLE TEST SOLUTION POLONIUM-210*

1. Public Service Company of Colorado, Fort St. Vrain Nuclear Station, Platteville, Colorado
2. General Electric Company, Vallecitos Nuclear Center, Pleasanton, California
3. U.S. Food and Drug Administration, Winchester Engineering and Analytical Center, Winchester, Massachusetts
4. Illinois Department of Public Health, Division of Laboratories, Springfield, Illinois
5. South Carolina Department of Health and Environmental Control, Division of Radiological Health, Columbia, South Carolina
6. Accu-Labs/CDM Limnetics, Wheatridge, Colorado
7. State Hygienic Laboratory of Iowa, Iowa City, Iowa
8. National Radiation Laboratory, Christchurch, New Zealand
9. Washington State Department of Social and Health Services, Environmental Laboratories Unit, Seattle, Washington
10. Michigan Department of Public Health, Division of Radiological Health, Lansing, Michigan
11. New York State Health Department, Radiological Sciences Laboratory, Albany, New York
12. Nebraska State Department of Health, Radiation Health Laboratory, Lincoln, Nebraska
13. Hazen Research, Inc., Golden, Colorado
14. Connecticut State Health Laboratories, Hartford, Connecticut
15. U.S. Environmental Protection Agency, Eastern Environmental Radiation Facility, Montgomery, Alabama
16. Wisconsin State Division of Health, Department of Health and Social Services, Madison, Wisconsin
17. Vermont State Health Department, Divison of Occupational Health, Barre, Vermont
18. Los Alamos Scientific Laboratory, H-5 Division, Los Alamos, New Mexico
19. University of Washington, College of Fisheries, Laboratory of Radiation Ecology, Seattle, Washington
20. Eberline Instrument Corporation, Albuquerque, New Mexico
21. Emory University, Physics Department, Atlanta, Georgia
22. Health and Welfare Canada, Radiation Protection Bureau, Ottawa, Ontario, Canada
23. U.S. Energy Research and Development Administration, Health and Safety Laboratory, Analytical Chemistry Division, New York, New York
24. LFE Environmental Analysis Laboratories, Technical Services Department, Richmond, California

* The order in which participants are listed in this table does not correspond to the order in which results are listed in Table 2.

EXPERIMENTAL DETAILS

Each participant analyzed the test solution by the method(s) of his choice. Some of the variations in the methods used are described in four categories.

DETECTORS USED

Six laboratories used internal gas flow proportional counters, nine laboratories used gas flow proportional counters, three laboratories used silicon surface barrier detectors, two laboratories used alpha-particle scintillation detectors, and one laboratory used a Frisch grid alpha-particle spectrometer. One laboratory used two detectors. Four laboratories did not indicate the detector used.

DILUTION OF SOLUTION

Nine laboratories did not dilute the solution and ten laboratories did dilute the solution. Five laboratories gave no indication of their preparation.

PREPARATION OF SOLUTION

Twelve laboratories prepared their solutions for counting by evaporating them on planchets, three laboratories prepared their solutions by electroplating the polonium onto planchets, two laboratories absorbed the polonium on silver disks, three laboratories precipitated polonium from their solutions and counted the filters, one laboratory used a hydrogen gas disposition, and three laboratories did not indicate how they prepared their counting sources.

CALIBRATION STANDARDS USED

Six laboratories indicated they used more than one radionuclide to calibrate their counting instrument. Fourteen laboratories indicated they used only one radionuclide. Four laboratories did not indicate which, or how many, radionuclides were used to calibrate their counting instruments. Four laboratories used polonium-210 sources, three laboratories used lead-210, four laboratories used plutonium-239, six laboratories used americium-241, two laboratories used gadolinium-148, two laboratories used thorium-230, two laboratories used polonium-208, one laboratory used neptunium-237, and one laboratory used plutonium-240.

RESULTS

The results reported by the 24 participants are given in Table 2 and Figure 1 as ratios of the participant's reported value of activity, corrected for decay to the date of calibration, to the NBS-calibrated activity value. This ratio is denoted as (x/NBS). For each x/NBS ratio, Figure 1 shows the linear sum of the random counting error at the 99-percent confidence level plus the linear sum of the estimated limits of systematic errors (as bars) as reported by the participant.

TABLE 2. RESULTS OF INTERLABORATORY INTERCOMPARISON OF POLONIUM-210 MEASUREMENTS

Participant	x/NBS	Uncertainty (%)	Participant	x/NBS	Uncertainty (%)
1	1.00	± 3.3	17	0.91	*
2	0.60	*	18	0.48	+ 21, - 9
3	0.91	± 8	19	1.02	± 4.6
4	0.98	± 1.7	20	1.02	± 13
5	0.62	*	21	0.28	*
6	0.36	± 7	22	0.33	± 1.8
7	0.96	± 2.3	23	0.82	± 7.7
8	0.76	± 10	24	0.47	± 10
10	0.95	± 6.1	25	0.95	± 1
14	1.37	± 3.6	26	1.02	± 28
15	1.04	± 4	27	1.01	± 7.6
16	0.96	± 10	28	0.76	± 7.2

* Not determinable

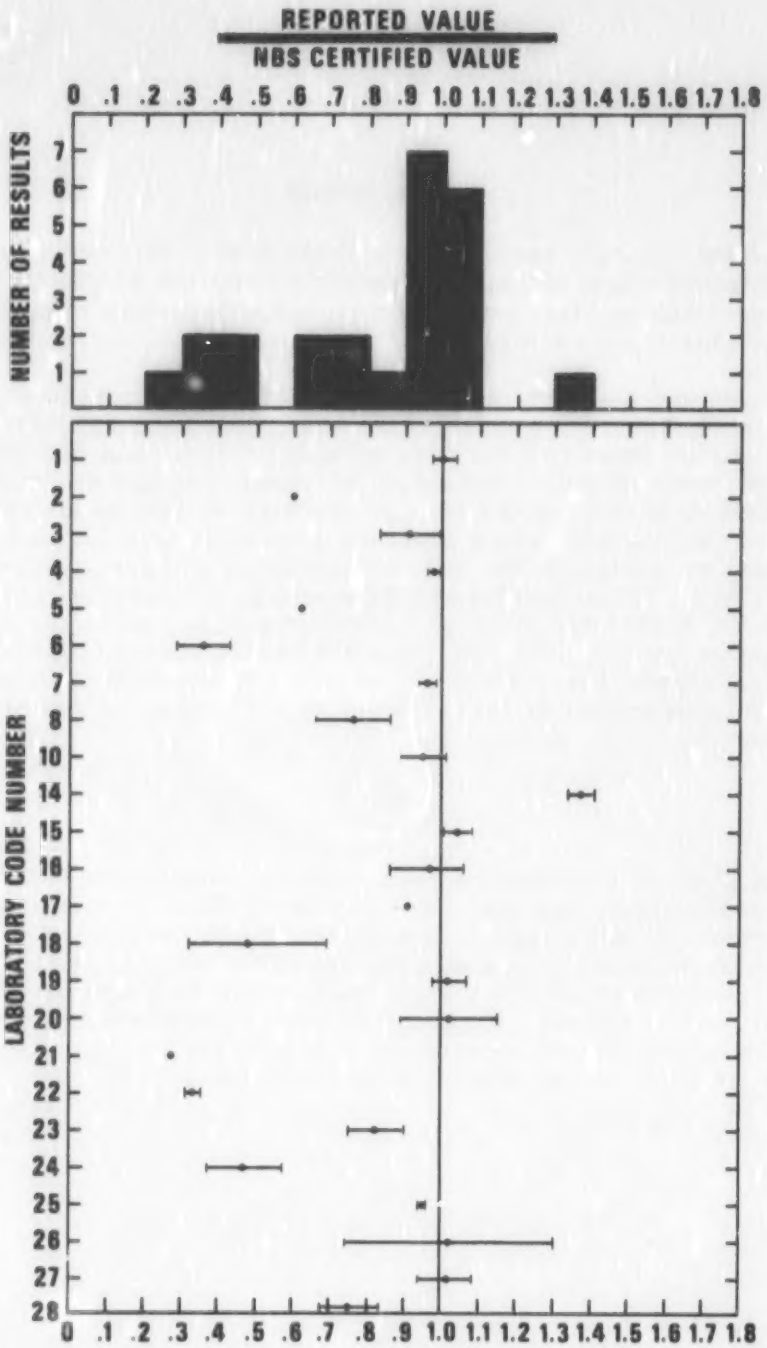

Figure 1. Reported results for the polonium-210 radioactivity test solution. Participants in this intercomparison are listed in Table 1. The bars are total estimated uncertainties (random plus estimated systematic errors). Uncertainties could not be determined for laboratories 2, 5, 17 and 21.

DISCUSSION

The x/NBS activity ratios have a range from 0.28 to 1.37. Thirteen of the participants reported values within ± 10 percent of the certified value of activity. Ten of the eleven who reported values more than 10 percent from the known value reported values that were lower than the known value.

Averages and standard deviations for each of the previously noted differences in methodology were computed (e.g., those who diluted their test solutions versus those who did not, as well as those who did not indicate dilution or nondilution). Inspection of these averages and standard deviations showed nothing significant. It was felt by the author that the sample size was so small that further statistical testing was not warranted. As a group, those participants who did not indicate complete experimental details and who filled out the test report form sloppily, tended to have values much below the NBS value for activity. Eight of the 24 participants considerably underestimated the magnitude of their systematic errors. There is no obvious correlation between total estimated errors and agreement with the NBS value. The bias for low values of activity may have been due to the high volatility of polonium (5).

SUMMARY

A total of 24 laboratories, representing power reactors, industry, State health organizations, national laboratories of foreign countries, Federal laboratories, and environmental consultant groups tested their measurement techniques on samples containing polonium-210. Only 54 percent of the reported results fell within ± 10 percent of the corresponding NBS values. These results are similar to the results of an interlaboratory intercomparison of a strontium-89, strontium-90-yttrium-90 radioactivity test source conducted by the National Bureau of Standards (6).

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APPENDIX

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
WASHINGTON, D.C. 20234

REPORT OF CALIBRATION

Alpha-Particle Test Solution

Polonium-210

prepared for

U. S. Environmental Protection Agency

Las Vegas, Nevada

This test solution consists of carrier-free polonium-210 in approximately 3.3 grams of approximately 2 molar nitric acid in a flame-sealed borosilicate-glass ampoule.

The number of alpha particles emitted per second per gram of solution at 1200 EST September 23, 1975, was

164.4 ± 0.9%.

This test solution was calibrated by means of liquid-scintillation counting. Confirmatory measurements were performed on sources that were deposited and dried on platinum discs, using the NBS 0.8π defined-solid-angle alpha-particle counter. The mean of these agreed with the calibration value to within 0.1 percent.

The uncertainty in the alpha-particle-emission rate, 0.9 percent, is the linear sum of 0.1 percent, which is the limit of the random error at the 99-percent confidence level (2.70 S_m, where S_m is the standard error computed from 47 determinations), and 0.8 percent, which is the estimated upper limit of conceivable systematic errors.

A half life of 138.378 ± 0.007 days is suggested [a].

The alpha-particle spectrum of an evaporated source prepared from this test solution was examined over the energy region of 4.0 to 8.0 MeV with a silicon surface-barrier detector system, and no alpha-particle-emitting impurities were detected. The detection limits for alpha particles from contaminants can be expressed as a percentage of the alpha-particle-emission rate of polonium-210 on the calibration date. For contaminant alpha particles with energies less than 5.3 MeV the detection limit would be approximately 0.1 percent, and for those with energies greater than 5.3 MeV the detection limit would be approximately 0.01 percent.

The gamma-ray-emission rate due to contaminants in this test solution over the energy region of 0.01 to 1.85 MeV is estimated to be less than 2 gamma rays per second per gram of solution on the calibration date.

The emission rate from this test solution of beta particles with energies greater than 0.15 MeV is estimated to be less than 2 beta particles per second per gram of solution on the calibration date.

(a) M. B. Lewis, Nuclear Data Sheets, B5(6), 631 (1971).

For the Director,

W. B. Mann

W. B. Mann, Chief
Radioactivity Section
Center for Radiation Research

November 21, 1975